IS 11007 (1984): Methabenzthiazuron, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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Indian Standard

SPECIFICATION FOR METHABENZTHIAZURON, TECHNICAL

UDC 632·954 METHABENZTHIAZURON

INDIAN STANDARDS INSTITUTION
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SPECIFICATION FOR METHABENZTHIAZURON, TECHNICAL

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Indian Standard

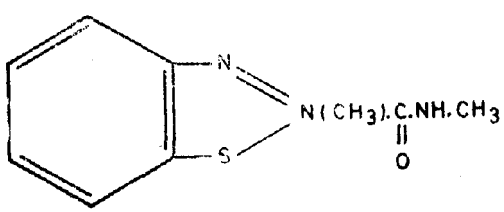
SPECIFICATION FOR METHABENZTHIAZURON, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 August 1984, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Methabenzthiazuron, technical is used for general weed control.

0.3 Methabenzthiazuron is the accepted common name by the International Organization for Standardization (ISO) for 1-(2-benzothiazolyl)-1-3-dimethyl urea. The empirical and structural formulae and molecular mass of methabenzthiazuron are indicated below:

Empirical Formula	*Structural Formula*	*Molecular Muss*
$C_{10}H_{11}N_3OS$		221.29

0.4 In the preparation of **this** standard, due consideration has been given to the provisions of the ***Insecticides Act,*** 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the act, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for methabenzthiazuron, technical.

2. REQUIREMENTS

2.1 Description — The material shall essentially comprise methabenzthiazuron and shall be in the form of white to pale yellow fine powder.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR METHABENZTHIAZURON, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix of this Standard	Cl No. of IS : 6940-1982*
(1)	(2)	(3)	(4)	(5)
i)	Methabenzthiazuron content, percent by mass, *Min*	90·0	A	—
ii)	Melting point, °C	108 to 115	—	6
iii)	Water Content, percent by mass, *Max*	1·0	—	4
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	0·2	—	11.3.2
	or			
	Alkalinity (as NaOH), percent by mass, *Max*	0·1		11.3.3

***Methods of test for pesticides and their formulations (*first revision*).**

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean and dry, mild steel drums or tinplate containers or fibreboard containers or double hessian jute bags (see IS : 8115-1976*) or DW tarpaulin laminated jute bags (see IS : 8117-1976†) or HDPE woven sacks [see IS : 8069 (Part 2)-19762] with a polyethylene liner of thickness not less than 0·062 mm. The containers shall also comply with the general requirements stipulated in 2 of IS : 8190 (Part 1)-1980§.

*Specification for **double hessian jute bags for pesticides.**

†Specification **for DW tarpaulin laminated jute bags for pesticides.**

#Specification for high density polyethylene (HDPE) woven sacks for packing pesticides: Part 2 Woven bags (first revision).

§Requirements **for packing of pesticides: Part 1 Solid pesticides** (*first revision*).

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to any other information as required under *Insecticides Act* and Rules:

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture;

d) Batch number;

e) Net mass of contents;

f) Methabenzthiazuron content, percent (*m/m*); and

g) The minimum cautionary notice as worded in *Insecticides Act* and Rules.

3.2.1 The container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. IS1 marked products are also continuously checked by IS1 for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in IS :10946-1984*.

5. TESTS

5.1 Tests shall be carried out by the methods as referred to in col 4 and 5 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (see IS :1070-1977†) shall be employed in tests.

NOTE — ' **Pure** chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods for sampling of technical grade pesticides.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

[Table 1, *Item* (i)]

DETERMINATION OF METHABENZTHIAZURON CONTENT

A-O. GENERAL

A-0.1 For the determination of methabenzthiazuron content, two methods, namely the infra-red spectrophotometric method and the UV-spectrophotometric method have been prescribed. Either of these methods may be used, but the method employed should be stated while expressing the result of a test.

A-l. INFRA-RED SPECTROPHOTOMETRIC METHOD

A-l.1 Apparatus

A-1.1.1 *Infra-red Spectrophotometer-* Capable of reading in the region of 1 800 cm^{-1} to 1 600 cm-l, with the slit width, gain and response time and scaning speed adjustable to produce satisfactory signal-to-noise ratio and adequate resolution under the conditions of the test.

A-1.1.2 *Absorption Cells* — potassium bromide cells having internal light path of about 0·15 mm.

A-1.1.3 *Hypodermic Syringe*

A-1.2 Reagents

A-1.2.1 *Methabenzthiazuron* — standard of known purity.

A-1.2.2 *Methylene Chloride* — pure.

A-l.3 Procedure

A-1.3.1 *Calibration*

A-1.3.1.1 Weigh accurately 0·45 g, 0'475 g, 0·50 g, 0·525 g and 0·55 g of methabenzthiazuron to the nearest 0·1 mg in 50-ml volumetric flask to give 0·9, 0·95, 1·0, 1·05 and 1'1 percent solution respectively. Dissolve and make up the volume with methylene chloride.

A-1.3.1.2 Fill the absorption cell with methylcne chloride. Adjust the spectrophotometer for the optimum instrument setting with respect to gain, slit width, response, speed and drum drive. Make scan over the wave length region 1 800 cm^{-1} to 1 600 cm^{-1} at least thrice and record the peak (I_o).

A-1.3.1.3 Turn the wave length scale back to its original setting. Fill the cell with solution one by one (see A-1.3.1.1). Make a scan in the wave length region of 1 800 cm^{-1} to 1 600 cm^{-1}, with the same setting as previously used (*see* A-1.3.1.2) and record the carbonyl peak (I_x).

A-1.3.1.4 Calculate the log I_0/I_x for each peak separately and find the average of 3 or 4 peaks. plot the graph log I_0/I_x versus percent concentration.

A-1.3.2 *Estimation of Active 'Ingredient in the Material* — Weigh about 0·50 g of the sample accurately nearest to 0·1 mg in a 50 ml volumetric flask. Dissolve in methylene chloride and make up to volume.

A-1.3.2.1 Make a scan of methylene chloride and methylene chloride soiution of the material (*see* A-1.3.2) as prescribed in A-1.3.1.2 and A-1.3.1.3 using the same cell and setting. Measure the peak I_0 and I_x respectively. Calculate the log I_0/I_x for each peak and take the average of the 3 peaks.

A-1.3.2.2 Read the value on the previously plotted graph and find the percent concentration.

A-l.4 Calculation

$$\text{Methabenzthiazuron content, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass as calculated from the graph, and

M = mass of sample taken for the test.

A-2. UV-SPECTROMETRIC METHOD

A-2.0 Principle — The active ingredient is separated from the technical or from the formulations by TLC on silica gel. The corresponding visible zone under UV-light is eluted with methanol. The methanolic extracts are measured UV-spectroscopically evaluating the absorption band at the longest wave length at 293 nm.

A-2.1 Apparatus

A-2.1.1 *TLC*

A-2.1.2 *UV-Visible Spectrophotometer* — with hydrogen source, range 360-280 nm, slit normal or programmed.

A-2.2 Reagents

A-2.2.1 *Methanol* — Spectroscopic grade.

A-2.2.2 *Methabenzthiazuron* — Chromatographically pure and of known purity.

A-2.2.3 *Silica Gel* — Chromatographic grade.

A-2.2.4 *Chloroform* — Analytical reagent grade.

A-2.3 Standardization

A-2.3.1 *Preparation of the Standard Solution* — Weigh 0·35 *g of pure* methabenzthiazuron to the nearest 0·1 mg into a 100 ml measuring flask, dissolve with chloroform. Dilute to volume with chloroform and shake thoroughly.

A-2.3.2 *Thin-Layer Chromatography* — Spot on the bottom of the plate 250 µl of the solution by pipette along the start line of a 20 x 20 cm plate charged with a 0·25 mm layer of silica gel. Develop the plate in a solvent system of chloroform : methanol (100 : 1). For drying, leave the plate for 10 minutes in the open air. Mark the main zone of the active ingredient which will appear as quenched fluoroscence under the UV-light of a wave length of 254 nm. Scrape off, thoroughly, the layer in the marked range and fill it quantitatively using a rubber wipper into a chromatographic column (length : 30 cm; i.d. : 1·5 cm) plugged with a wad of cottonwool. Elute the active ingredient with 95 ml of spectroscopically pure methanol into an 100 ml measuring flask, dilute to volume with the same methanol and shake *vigorously*. Total dilution volume '*V*' shall be equal to 40 000 ml.

A-2.3.3 *UV-spectroscopic Measurement* — Fill into a matched pair of 1·0 cm cells the clear standard solution and spectroscopically pure methanol. Record the spectrum in the range of 360-280 nm. Read the absorbance of the maximum at 293 nm according to the corrected base line method.

A-2.4 Analysis

A-2.4.1 *Preparation of the Analysis Solution*

A-2.4.2 Weigh an amount of sample containing about 0·35 g of the technical active ingredient to the nearest 0·1 mg into a 100-ml measuring flask, add 80 ml of chloroform, warm for half an hour on a water-bath, allow to cool, dilute to volume with chloroform and shake vigorously.

A-2.4.3 Carry out TLC as described in A-2.3.2 followed by eluting through chromatographic column and making the dilution volume '*V*' equal to 40 000 ml.

A-2.4.4 Measure the absorbance of solution (see A-2.4.3) as mentioned in A-2.3.3 in a matched pair of 1·0 cm cells against spectroscopically methanol.

A-2.5 Calculation

Methabenzthiazuron content, percent by mass = $\frac{A_1}{A} \times \frac{M}{M_1} \times P$

where

A_1 = absorbance for sample solution;

M = mass, in g, of standard methabenzthiazuron taken for the test;

P = purity of the standard methabenzthiazuron;

A = absorbance for standard solution; and

M_1 = mass, in g, of sample taken for the test.

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